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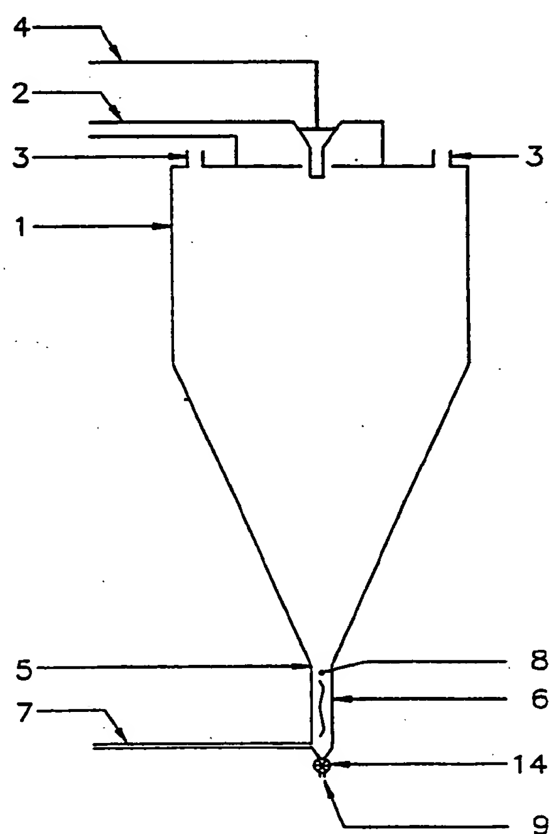
(54) Title: PROCESS AND APPARATUS FOR PRODUCING A GRANULATE BY SPRAY DRYING

(57) Abstract

A substantially dust-free granulate with a desired mean particle size and a narrow particle size distribution is produced by a spray drying process comprising atomizing a solution or suspension of the material to be granulated to droplets; contacting said droplets with a stream of drying gas and a stream of solid feed particles in a spray drying and granulation zone to obtain a granulate by collision between droplets and feed particles and between moist feed particles; withdrawing the particles or granulates from the spray drying and granulation zone; and subjecting these particles or granulates to a size classification into two fractions in a countercurrent gas/gravity classifier, comprising a coarse fraction of the predetermined particle size which is withdrawn as product and an undersize fraction, which is returned to the spray drying and granulation zone as solid feed particles.



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Process and apparatus for producing a granulate by spray drying

The present invention relates to a process and an apparatus for producing a substantially dust-free granulate with a desired mean particle size and a narrow particle size distribution by spray drying.

In the present context "granulation" is defined as the building up of larger solid particles from smaller solid particles, also referred to as "(fine) solid starting material/particles"; "granulate" and "granulate material" is defined as the product obtained by granulation.

It is well known that granulate material may be produced by using fluidized bed methods where a solution or suspension of the material to be granulated is sprayed on or into a fluidized bed containing solid particles to be granulated. It is also well known that the finer part of the product discharged from the fluidized bed may be returned for further granulation in the fluidized bed, cf. DEOS 22 31 445; DEOS 25 55 917; EP 87 039 and EP 163 836.

A typical feature of the fluidized bed methods consists in that the granulation process is taking place under conditions corresponding to a completely stirred reactor. Therefore, these methods operate with long residence times for the solid particles. The solid starting material is substantially identical with the material discharged from the granulation zone, and it is thus generally necessary to separate the discharged material into thr e fractions, viz. a fine undersize fraction, a product fraction and an oversize fraction, which may be ground and r turned to the granulation zone.

The term "granulation efficiency" is defined as the weight fraction of material leaving the granulation zone which falls within a specified, desired product range. The "recycle ratio" is defined as the weight ratio between recirculated material and material withdrawn as product.

Until now no efficient fluidized bed process has been disclosed yielding a dust-free product with desired mean particle size and a narrow particle size distribution.

It is further known that the products produced by ordinary spray drying generally have a low mean particle size, typically within the range 50-150 µm, in some cases even less and often present serious dust problems.

In order to produce larger sized particles utilizing the principles of spray drying combined spray drying, fluidized bed and agglomeration processes have been suggested.

From US PS 3,849,233 and EP 97 484 a process is known wherein a fluidized bed is arranged at the lower part of the drying chamber of a spray drying unit. In this process the particles produced in the spray drying zone are blown into the fluidized bed by the drying gas from the spray drying zone. Hereby particles from the fluidized bed are blown up into the spray drying zone. Since the spray drying zone is operated to provide exit particles with a rather high moisture content agglomeration may take place in the spray drying zone as well as in the fluidized bed.

From US PS 3,735,792 another combined spray drying fluidized b d and agglomeration process is known. This process takes also place in a chamber containing a fluidized bed arranged at the bottom of said chamber. Liquid f ed is spray d as droplets into the chamber by a

fluid atomizer arranged at the top of the chamber. Primary air enters through a perforated plate at the bottom of the chamber and acts as fluidization and drying air. Secondary air fed in at the top of the chamber imparts a spiral motion to the air flow in the chamber whereby particles are withdrawn from the upper part of the fluidized bed and suspended particles are caused to make a circulatory motion in the zone above the fluidized bed. Granulates are thus formed by collision between particles and droplets and between moist particles, presumably mainly in the fluidized bed. Particles are continuously discharged from the fluidized bed, preferably via a discharge pipe countercurrently to a rising air stream, which may return a certain amount of the fines in the withdrawn product to the fluidized bed whereby a limited reduction of the dust content of the product may be achieved.

Finally, the so-called straight-through process should be mentioned as a combined spray drying and granulation process. By this process the product withdrawn from the spray drying zone is subjected to a further drying step in a fluidized bed. Particles removed from the fluidized bed by the fluidizing gas are returned to the spray drying zone and granulation takes place in this zone by collision between return particles and droplets and between moist return particles.

It is important to observe that the particle build-up mechanisms in a fluidized bed process differ from that of a spray drying granulation process. The most essential differences being the following:

As mentioned above the fluidiz d bed granulation process corresponds to a reaction in a completely stirr d reactor having a long residence time for the reactor content, wherein the solid starting material, as defined above, is

substantially identical with the material discharged from the granulation zone, whereas the process comprising granulation in the spray drying zone is similar to the reaction in a plug flow reactor with a very short residence time for the reactants, wherein the solid starting material is substantially identical with the recirculated material.

In the fluidized bed granulation process only a minor amount of liquid is introduced per second relative to the solid material available, whereas a much larger specific amount of liquid is introduced in the spray drying granulation process. It is desired to operate with a high liquid solid ratio in the granulation zone because this favours high strength of the granulates. However, fluidized bed granulation process cannot be safely operated at such high ratios, because the process becomes increasingly unstable, when the liquid solid ratio is increased, the problem being an increasing risk for break down of the granulation process due to formation of large lumps of sticky material.

In all granulation processes it is necessary to introduce or create fine starting particles necessary for the build-up of the granulated particles. In the fluidized bed granulation process these fine starting particles are mainly generated by attrition or grinding of already formed granulates and the rate of introduction of these fine particles into the granulation process can only be effectively controlled by controlled introduction of ground granulates, e.g. oversize particles. In contrast, fine starting particles are easily produced in the spray drying agglomeration by evaporation of droplets which have not collid d with solid particles in the spray drying zone.

In the fluidized bed granulation process the starting

particles are generally only comprising a small percentage of fine particles, whereas a large percentage of fine starting particles is characteristic for the known granulation processes involving spray drying. This high percentage is actually representing a problem in the straight-through process because it leads to the formation of a granulated product with an unacceptably high content of dust.

Granulates produced in a fluidized bed are in general rather coarse with a typical mean particle size about 1-5 mm, whereas granulates produced in a spray drying zone in general have a mean particle size less than about 300 µm.

For environmental reasons the demand for a low dust content in solid products has increased considerably during recent years, not least in respect of products for use in agriculture and the dyestuff industry. These demands have only with great difficulty been satisfied in respect of products produced according to the known art.

The object of the invention is to provide a process and an apparatus for producing a granulated product

- with a low dust content;
- with a desired mean particle size (d_{50});
- with d_{50} value smaller than or equal to about 1000 µm, preferably within the interval 150-400 µm;
- with a narrow particle size distribution corresponding to at least 70, preferably at least 80 % by volume, between 0.5 d_{50} and 1.5 d_{50}; and
- having high abrasion strength and excellent fluid-mechanical properties;

by a spray drying granulation process in which

- the recycle ratio is kept at a very low level;
- the mass ratio between liquid and solid feed to the spray drying and granulation zone is high;
- the formation of dust is reduced to a minimum;
- the residence time of the material in the granulation zone is kept at a very low level; and
- a subsequent cooling of the granulated product can be performed in a highly efficient way;

which method can be carried out in/which apparatus is a compact apparatus which, if desired, may be constructed by slight and inexpensive modification of existing spray drying plants.

We have now found that the object of the invention set forth above can be attained by a process for producing a substantially dust-free granulate with a desired mean particle size and a narrow particle size distribution by spray drying a solution or suspension of the material to be granulated comprising the steps of

atomizing the solution or suspension to droplets in an atomizing zone in the upper part of a spray drying and granulation zone in a drying chamber;

contacting said atomized solution or suspension with a central downward stream of drying gas and a stream of solid feed particles introduced into the upp r part of the spray drying and granulation zone to obtain a granulate by enlargement of the size of said particles by

collision between droplets and feed particles and between moist feed particles;

adjusting the amount of introduced solution or suspension to the amount and drying capacity of introduced drying gas to ensure a desired moisture content of the particles or granulates leaving the spray drying and granulation zone and evaporation of non colliding droplets to formation of fine particles;

withdrawing a stream of spent drying gas from the spray drying and granulation zone;

withdrawing the particles or granulates leaving the spray drying and granulation zone from the drying chamber; and

subjecting said withdrawn particles or granulates to a size classification into two fractions in a counter-current gas/gravity classifier, preferably multistage, wherein the particles or granulates are introduced in a rising gas stream in a separation zone and separated into a coarse fraction of the predetermined particle size which is withdrawn as product and an undersize fraction, which is withdrawn from the separation zone as an entrained suspension in the exit gas from the classifier and returned to the spray drying and granulation zone as solid feed particles.

By this method it is possible to produce a substantially dust-free granulated product with a d_{50} value within the desired range and the desired narrow particle size distribution having high abrasion strength and excellent fluid-mechanical properties.

The granulate obtained by the method according to the inventi n may typically exhibit a mean particle size within the interval 150-400 µm; a particle size

distribution showing at least 70, typically at least 80 % by volume particles in the interval [0.5 d_{50}, 1.5 d_{50}] and exhibit a low content of dust.

The recycle ratio is low, the mass ratio between liquid and solid feed to the spray drying and granulation zone is high and the residence time of the material in the granulation zone may be kept at a very low level, which is particularly advantageous when granulating heat sensitive materials.

One of the characteristic features of the invention resides in the fact that the use of a countercurrent gas/gravity classifier allows a very "sharp cut" as defined below:

The efficiency of a particle size classifier may be expressed by the so-called grade efficiency curve which is a graph showing g(x) as a function of x, where x is particle size and g(x) is the volume percent of particles separated as coarse particles in the size range [x,x+dx].

A classifier exhibiting a "sharp cut" is defined as a classifier with a low value for x_{90}/x_{10}, typically less than 6.0; where x_y is defined by $g(x_y) = y$. Counter-current gas/gravity classifiers exhibiting x_{90}/x_{10}-values less than 6.0 are obtainable in practice, and x_{90}/x_{10}-values less than 4.2 and even less than 2.5 can also be achieved by these classifiers.

An essential advantage of using a countercurrent gas/gravity classifier resides in that the cut size, defined as the value of x_{50}, may be varied within a rather broad range, e.g. by adjusting the gas velocity in the classifier, typically b tween 0,3 and 3 m/s, independently of the other process parameters. A selected mean particl size of the granulate product is obtained by

selecting a suitable value for the cut size of the classifier.

A further essential advantage of using a countercurrent gas/gravity classifier consists in the "sharp cut" obtained by using this type of classifier, which ensures an extremely favourable particle distribution of the stream of solid feed particles introduced into the upper part of the spray drying and granulation zone, which, as mentioned above, is substantially identical with the particle distribution of the solid starting material in the granulation process according to the invention.

Since only a minor amount of coarse particles are re-circulated to the granulation zone not only the desired narrow particle size distribution of the product, but also the desired granulation efficiency are obtained.

Another characteristic feature of the countercurrent gas/gravity classifier resides in the possibility of operating with a relatively small gas volume compared with the gas volume used in a far less efficient fluid-ized bed classifier.

Since the process according to the invention operates with a high ratio between the liquid feed and fine solid starting material high granulate strength is obtained.

The solution or suspension may be atomized to droplets in a rotary atomizer, a pressure nozzle atomizer or a pneumatic or two-fluid atomizer.

The exit gas from the spray drying granulation zone may entrain fine particles from the granulation zone. If desir d, these "exit gas particles" may be precipitated, e.g. in a cyclone, and returned to the spray drying granulation zone, preferably to the upper part of that

zone.

The recirculated particles from the classifier and the exit gas particles if returned are preferably introduced into the spray drying granulation zone in the immediate vicinity of the atomizing zone.

The moisture content of the particles or granulates leaving the spray drying and granulation zone is preferably adjusted to a value ensuring that said particles or granulates do not exhibit any sticky character. This measure is particularly important when the process does not comprise further drying in a second drying zone in a fluidized bed.

This process is suited for agglomerating a large number of materials including

agrochemical compounds such as pesticides, e.g. insecticides, acaricides, nematodicides and fungicides, herbicides, plant growth controlling compounds and fertilizers;

pharmacologically active compounds;

animal feed products;

food products including milk products;

dyestuffs and pigments;

resins and polymers;

and other organic and inorganic substances including fermentation products, plant extracts, amino acids and proteins, enzymes, food additives, and byproducts from the slaughterhouse, dairy and paper and pulp industry.

The process is particularly suited for granulating heat sensitive materials.

If desired, the particles or granulates leaving the spray drying and granulation zone may be subjected to further drying in a second drying zone comprising a fluidized bed arranged at the bottom of the drying chamber before they are withdrawn from the drying chamber.

If desired, further granulation may be achieved in the fluidized bed, e.g. by spraying a minor amount of liquid into or onto the fluidized bed.

According to a preferred embodiment a multistage countercurrent gas/gravity classifier is employed, preferably a "zigzag classifier". The construction and operation principles of zigzag classifiers are well known and described in e.g. GB PS 468 212.

When the process according to the invention is carried out without a second drying zone in a fluidized bed at the bottom of the drying chamber the undersize fraction from the classifier may be reintroduced via a particle exit opening at the bottom of the spray drying agglomeration chamber and blown directly into the upper part of the spray drying and granulation zone by the exit gas from the classifier.

However, improved contact between droplets and recirculated material may be achieved in an embodiment where the particles or granulates leaving the spray drying and granulation zone are withdrawn from the drying chamber via a gas lock and the undersize fraction from the classifi r is transported to the upper part of the spray drying and granulation chamber entrained by the exit gas from the classifier and introduced into said

upper part, optionally after having been separated from said exit gas.

When the process according to the invention is carried out with a second drying zone in a fluidized bed at the bottom of the drying chamber the particles or granulates may be withdrawn through an exit opening situated at the upper part of the fluidized bed, the undersize fraction being reintroduced via said exit opening and blown directly into the upper part of the spray drying and granulation zone by the exit gas from the classifier.

However, improved contact between droplets and re-circulated material may be achieved in an embodiment wherein the particles or granulates are withdrawn from the lower part of the fluidized bed via a gas lock or through an exit opening situated at the upper part of the fluidized bed and the undersize fraction from the classifier is transported to the upper part of the spray drying and granulation chamber entrained by the exit gas from the classifier and introduced into said upper part, optionally after having been separated from said exit gas.

According to a preferred embodiment the cut size of the air classifier is selected within the interval 1.2-5.5 times the upper limit for the dust interval, defined as the size range for unacceptable fines in the product, preferably in the interval 2.0-3.5 times the said upper limit.

According to another preferred embodiment the recycle ratio is selected within the range 0.15-3, preferably within the range 0.2-1.0.

According to a further preferred embodiment the classi-fier is adjusted to provide a separation efficiency

corresponding to a x_{90}/x_{10}-value (as defined above) less than 6.0, preferably less than 4.2, in particular less than 2.5.

According to a preferred embodiment the countercurrent gas/gravity classifier is utilized as a cooler by introducing a cold gas into the classifier.

This embodiment is particularly advantageous because of a very efficient countercurrent heat exchange, resulting in a substantial reduction of the volume of cooling gas, and a very compact apparatus, compared to the alternative: a fluidized bed cooler.

The process may be carried out in an apparatus for producing a substantially dust-free granulate with a desired mean particle size and a narrow particle size distribution by spray drying a solution or suspension of the material to be granulated comprising a spray drying granulation chamber with substantially conical, downward tapering walls provided with

a drying gas inlet, and a liquid inlet provided with an atomizer, arranged in the upper part of the chamber;

a drying gas outlet;

an inlet for a stream of solid feed particles;

a material outlet arranged at the lower part of the chamber connected to a material inlet of a countercurrent gas/gravity classifier with a separation gas inlet, a product outlet provided with a gas lock and an outlet for a fraction of fine particles suspended in separation gas conn cted to the inlet for the stream of solid feed particles.

According to a preferred embodiment the material outlet of the chamber is connected to the material inlet of the classifier via a gas lock, and the inlet for the stream of solid feed particles is arranged in the upper part of the chamber and connected to the fine material fraction outlet of the classifier via a conduit.

According to another preferred embodiment a perforated horizontal plate for supporting a fluidized bed and an inlet for fluidizing gas are arranged at the bottom of the chamber.

According to a further preferred embodiment an opening functioning as material outlet of the chamber as well as inlet for the stream of solid feed particles is arranged at the lower part of the chamber at a position over the perforated horizontal plate for supporting the fluidized bed.

According to yet a further preferred embodiment the material outlet of the chamber is arranged at the perforated horizontal plate for supporting the fluidized bed and connected to the material inlet of the classifier via a gas lock or as an opening arranged at the lower part of the chamber at a position over the perforated horizontal plate for supporting the fluidized bed and connected to the material inlet for the classifier; and the inlet for the stream of solid feed particles is arranged in the upper part of the chamber and connected to the fine material fraction outlet of the classifier via a conduit.

The invention is further described with reference to the drawing illustrating the present invention.

On th drawing

- figure 1 and 2 show schematically lay-outs for a spray

drying granulation process without fluidized bed;

- figure 3 and 4 show schematically lay-outs for a spray drying granulation process comprising a fluidized bed;

- figure 5 and 6 are photomicrographs of particles of protein produced by the process according to the invention and ordinary spray drying, respectively; and

- figure 7 shows a graph of d_{50} as a function of the gas velocity, v, in the air classifier.

In fig. 1 1 represents a spray drying granulation chamber provided with a drying gas inlet 2, two drying gas outlets 3, a drying gas outlet may also be arranged at the lower part of the chamber 1, and a liquid inlet 4 provided with an atomizer. The chamber 1 has a material outlet 5 connected to a countercurrent gas/gravity classifier 6 with a gas inlet 7, an outlet 8 for fines suspended in gas and a product outlet 9 provided with a rotary valve 14.

In operation hot drying gas is introduced via the drying gas inlet 2 and liquid is introduced into the chamber 1 via a liquid inlet 4 and atomized to droplets. A part of these droplets evaporate under formation of fine solid particles and a substantial part of these droplets collide with the fine particles recirculated from the classifier 6 under formation of granulates which are withdrawn with the fine solid particles from the granulation chamber via the material outlet 5 and screened in the air classifier 6. The granulated end product is discharged as the coarse fraction from the classifi r 6 via the product outl t 9. The spent drying gas from th spray drying granulation zone is withdrawn via the gas outlets 3. This gas may contain entrained fine particl s which, if desired, may be precipitated in

a cyclone or a filter and returned to the spray drying granulation zone.

In fig. 2 1 represents a spray drying granulation chamber and 2, 3, 4, 6, 7, 8, 9 and 14 refer to corresponding apparatus parts as above. In this embodiment the outlet 8 is connected to a transfer pipe 10 with an opening 11 situated in the immediate vicinity of the atomizing zone. The recirculated fine material may be precipitated in a not shown cyclone before it is introduced into the spray drying granulation zone. In this case the material outlet 5 is provided with a rotary valve 15.

In fig. 3 1 represents a spray drying granulation chamber provided with a fluidized bed 12 having a fluidization gas inlet 13 and arranged at the bottom of the chamber 1. Reference numerals 2, 3, 4, 5, 6, 7, 8, 9 and 14 refer to corresponding apparatus parts as above. But in this case the material outlets 5 and 8 are arranged at the upper part of the surface of the fluidized bed 12.

In operation heating gas is introduced via the heating gas inlet 2 and liquid is introduced into the chamber via the liquid inlet 4 and atomized to droplets. A part of these droplets evaporate under formation of fine solid particles and a substantial part of these droplets collide with the fine particles recirculated from the classifier 6 under formation of granulates. These granulates and the fine solid particles are transferred to the fluidized bed 12 where they are subjected to further drying. The material is withdrawn from the fluidized bed via the material outlet 5 and screened in the air classifier 6. The granulated end product is discharged as the coarse fraction fr m the classifier 6 via the product outlet 9. The spent heating gas from the spray drying granulation zone is withdrawn via the gas outlets 3. This gas may contain ntrained fine particles

which, if desired, may be precipitated in a cyclone and returned to the spray drying granulation zone.

In fig. 4 1 represents a spray drying granulation chamber provided with a fluidized bed 12 having a fluidization gas inlet 13 and arranged at the bottom of the chamber 1. Reference numerals 2, 3, 4, 5, 6, 7, 8, 9 and 14 refer to corresponding apparatus parts as above. But in this case the material outlet 5 is arranged at the bottom of the fluidized bed 12 and provided with a rotary valve 15, and the outlet 8 is connected to a transfer pipe 10 with an opening 11 situated in the immediate vicinity of the atomizing zone. Also in this case the recirculated fine material may be precipitated in a not shown cyclone before it is introduced into the spray drying granulation zone.

These embodiments are only given as examples. In another preferred embodiment the material outlet 5 is arranged as shown in fig. 3, whereas the outlet 8 of the classifier 6 is connected to a transfer pipe 10 with an opening 11 situated in the immediate vicinity of the atomizing zone as shown in fig. 4.

The process according to the invention will be further illustrated by means of the following examples.

EXAMPLE 1

A suspension of single-cell protein with a content of 13% dry matter was granulated in an apparatus according to the invention by means of a rotary atomizer. In this apparatus the material outlet 5 was arranged as shown in fig. 3, the outlet 8 of the classifier 6 was connected to a transfer pipe 10 with an opening 11 situated in the immediate vicinity of the atomizing zon as shown in fig. 4, and the gas outlet 3 was arranged not at the upper

part of th chamber, but at the lower part over the surface of the internal fluidized bed.

The amount of feed was approx 800 kg/h. The granulated material was via an internal fluidized bed in the chamber passed to a countercurrent gas/gravity classifier. The internal fluidized bed was used for afterdrying and distribution of the granulate to the classifier. The amount of drying air introduced into the spray drying chamber was approx. 7000 kg/h, and the amount of air for the classifier which was also used as a cooler (cold separation air) was 160 kg/h corresponding to a gas velocity in the classifier of 0.5 m/s. Fine particles from the air classifier as well as fine particles separated from the exit gas from the spray drying granulation chamber were recirculated to the atomizing zone by means of a pneumatic conveyor system.

Further process conditions and data are shown in table 1.

TABLE 1

Temperature	
Feed, °C	40
Inlet gas to chamber, °C	338
Gas to classifier, °C	21
In fluidized bed, °C	63
Exit gas from chamber, °C	87
RH*) in chamber, %	25
MC*) in product, %	4.1
Classifier properties	
x_{90}/x_{10} ratio	about 4
Cut size/upper dust limit ratio	about 2.6

*) RH: Relative humidity; MC: Moisture content

The granulat s withdrawn as product from the air

classifier had a temperature of 28°C and exhibit d a particle size distribution, which can be expressed as a Rosin-Rammler-distribution having a mean size of 157 μm and a slope of 3.2, corresponding to d_{50} = 140 μm, and 73% by vol. between 0.5 d_{50} and 1.5 d_{50}.

A scanning picture of these granulates is shown in fig. 5.

For reasons of comparison a suspension of the same starting material but with a content of 25% dry matter was spray dried in a corresponding ordinary spray dryer. Fig. 6 shows a scanning picture of the single-cell particles obtained as a product by this process. This product exhibited: d_{50}: 20 μm, and 66 % vol. between 0.5 d_{50} and 1.5 d_{50}.

The flowability-properties of these products were determined by measurement of the Hausner ratio.

As further described in e.g. Svarovsky, L., Powder Testing Guide, Elsevier (1987) the Hausner ratio is defined as the ratio of the tapped bulk density to the untapped bulk density.

A value of the Hausner ratio close to unity indicates weak interparticle forces ensuring excellent flowability, whereas a high value e.g. greater than 1.4 indicates poor flowability.

The Hausner ratio for the product produced according to the invention was measured to 1.06, and the Hausner ratio for the product produced by ordinary spray drying was measured to 1.46.

EXAMPLE 2

An aqueous suspension of an organic agricultural chemical with a content of 32% dry matter was granulated in an apparatus similar to the one used in example 1. However, the suspension was not atomized by means of a rotary atomizer, but by a nozzle with a working pressure of 70 bar, and the gas outlet was arranged in the upper part of the chamber as shown in fig. 3. A minor amount of the suspension was sprayed directly onto the internal fluidized bed by a secondary nozzle arranged immediately over the internal fluidized bed. Approx. 350 kg aqueous suspension were atomized per hour, 15% thereof through the secondary nozzle. The classifier was operated with an amount of air of 530 kg/h corresponding to a gas velocity of 1.5 m/s in the classifier.

Further process conditions and data are shown in table 2.

TABLE 2

Temperature	
Feed, °C	34
Inlet gas to chamber, °C	198
Gas to classifier, °C	21
In fluidized bed, °C	72
Exit gas from chamber, °C	87
RH*) in chamber, %	10
MC*) in product, %	6.5
Classifier properties	
x_{90}/x_{10} ratio	about 3
Cut size/upper dust limit ratio	about 2.6

*) RH: R lativ humidity; MC: Moisture content

Th resulting product was non-dusting and contained only 2% by volume particles of a size less than 125 µm. The

particle size distribution was characterized by: d_{50} = 495 µm, and 88% between 0.5 d_{50} and 1.5 d_{50}.

The flowability-properties of this product was determined by measurement of Hausner ratio: The Hausner ratio for the product produced according to the invention was measured to 1.11.

EXAMPLE 3

Sulfite liquor from a plant for treating cellulose by the sulfite process having a content of dry matter of approx. 50% was granulated in an apparatus similar to the one used in example 1. Between 600 and 700 kg of this liquor was atomized per hour.

Further process conditions and data are shown in table 3.

TABLE 3

Temperature	
Feed, °C	approx. 40
Inlet gas to chamber, °C	220
Gas to classifier, °C	23-26
In fluidized bed, °C	70
Exit gas from chamber, °C	94
RH*) in chamber, %	10
MC*) in product, %	4

*) RH: Relative humidity; MC: Moisture content

Three series of tests were carried out in which the classifier was operated at three different air velocities while the other process conditions were kept essentially constant. For each value of the air velocity the particle distribution in the resulting product was determined. The results show d that the particle size increased monoto-

nously with increasing air velocity in the classifier. The amount of small particles was also reduced when the air velocity was increased.

The results are shown in the following table 4 and in fig. 7:

TABLE 4

Air velocity m/s	Median diameter d_{50} µm	Amount less than 50 µm %
0.6	110	12
1.1	140	7
1.6	171	5

The flowability-properties of these products were determined by measurement of the Hausner ratio:

The Hausner ratios for the products produced according to the invention were measured to 1.22, and did not show any significant variation as function of the air velocity in the classifier.

EXAMPLE 4

Sulfate liquor from a plant for treating cellulose by the sulfate process having a content of dry matter of approx. 30% was granulated in an apparatus similar to the one used in example 1. Spray drying of sulfate liquor normally results in a product containing large amounts of dust.

Two tests were carried out. In the first test a rotary atomizer was used. In the second the feed was atomized by means of a nozzle having a working pressure of 80 bar.

In both tests approx. 400 kg of this liquor was atomized per hour.

Further process conditions and data are shown in table 5.

TABLE 5

Temperature	(rot.a./nozzle)
Feed, °C	approx. 60
Inlet gas to chamber, °C	170/175
Gas to classifier, °C	26
In fluidized bed, °C	66
Exit gas from chamber, °C	72/75
RH*) in chamber, %	19/17
MC*) in product, %	4
Classifier properties	
x_{90}/x_{10} ratio	about 3

*) RH: Relative humidity; MC: Moisture content

The air velocity in the classifier was 1.5 m/s corresponding to an amount of air of 520 kg/h.

Particle size distribution and flowability data for the products are shown in table 6. For reasons of comparison corresponding data for a product produced by ordinary spray drying are also shown in table 6.

TABLE 6

	Median diameter d_{50} µm	Amount less than 50 µm %	Hausner ratio
Test with rotary atomizer, I*)	144	3	1.21
Test with nozzle, I*)	160	2	1.19
Test with ordinary spray drying, C*)	32	76	1.36

*) I: According to the invention; C: Comparison (known art)

It appears from the table that the products produced by the method according to the present invention exhibit a considerably larger particle diameter, a lower content of dust and a lower Hausner ratio than the product produced according the known art. Moreover, there is only a minor difference between the product obtained by the use of a rotary atomizer and the product obtained by the use of a nozzle atomizer

C l a i m s :

1. A process for producing a substantially dust-free granulate with a desired mean particle size and a narrow particle size distribution by spray drying a solution or suspension of the material to be granulated comprising the steps of

atomizing the solution or suspension to droplets in an atomizing zone in the upper part of a spray drying and granulation zone in a drying chamber;

contacting said atomized solution or suspension with a central downward stream of drying gas and a stream of solid feed particles introduced into the upper part of the spray drying and granulation zone to obtain a granulate by enlargement of the size of said particles by collision between droplets and feed particles and between moist feed particles;

adjusting the amount of introduced solution or suspension to the amount and drying capacity of introduced drying gas to ensure a desired moisture content of the particles or granulates leaving the spray drying and granulation zone and evaporation of non colliding droplets to formation of fine particles;

withdrawing a stream of spent drying gas from the spray drying and granulation zone;

withdrawing the particles or granulates leaving the spray drying and granulation zone from the drying chamber; and

subjecting said withdrawn particles or granulates to a size classification into two fractions in a counter-current gas/gravity classifier, preferably multistage,

wherein the particles or granulates are introduced in a rising gas stream in a separation zone and separated into a coarse fraction of the predetermined particle size which is withdrawn as product and an undersize fraction, which is withdrawn from the separation zone as an entrained suspension in the exit gas from the classifier and returned to the spray drying and granulation zone as solid feed particles.

2. A process according to claim 1, wherein the particles or granulates leaving the spray drying and granulation zone are subjected to further drying in a second drying zone comprising a fluidized bed arranged at the bottom of the drying chamber before they are withdrawn from the drying chamber.

3. A process according to claim 1 or 2, wherein the countercurrent gas/gravity classifier is a multistage zigzag classifier.

4. A process according to claims 1 or 3, wherein the undersize fraction is reintroduced via a particle exit opening at the bottom of the spray drying agglomeration chamber and blown directly into the upper part of the spray drying and granulation zone by the exit gas from the classifier.

5. A process according to claims 1 or 3, wherein the particles or granulates leaving the spray drying and granulation zone are withdrawn from the drying chamber via a gas lock and the undersize fraction from the classifier is transported to the upper part of the spray drying and granulation chamber entrained by the exit gas from th classifier and introduced into said upper part, optionally after having been separated from said exit gas.

6. A process according to claims 2-3, wherein the particles or granulates are withdrawn through an exit opening situated at the the upper part of the fluidized bed and the undersize fraction is reintroduced via said exit opening and blown directly into the upper part of the spray drying and granulation zone by the exit gas from the classifier.

7. A process according to claims 2-3, wherein the particles or granulates are withdrawn from the fluidized bed via a gas lock or through an exit opening situated at the upper part of the fluidized bed, and the undersize fraction from the classifier is transported to the upper part of the spray drying and granulation chamber entrained by the exit gas from the classifier and introduced into said upper part, optionally after having been separated from said exit gas.

8. A process according to claims 1-7, wherein the cut size of the air classifier is selected within the interval 1.2-5.5 times the upper limit for the dust interval, defined as the size range for unacceptable fines in the product, preferably in the interval 2.0-3.5 times the said upper limit.

9. A process according to claims 1-8, wherein the recycle ratio, defined as the weight ratio between recirculated material and withdrawn product is selected within the range 0.15-3, preferably within the range 0.2-1.0.

10. A process according to claims 1-9, wherein the classifier is selected to provide a separation efficiency corresponding to a x_{90}/x_{10}-value less than 6.0, preferably less than 4.2, in particular less than 2.5, where x_y is the particl size for which $g(x_y)$ = y % by volume, wh re g( ) is the graph of the grade efficiency curve of

the classifier.

11. A process according to claims 1-10, wherein the product is subjected to cooling in the classifier by introducing a cold gas in the classifier.

12. An apparatus for producing a substantially dust-free granulate with a desired mean particle size and a narrow particle size distribution by spray drying a solution or suspension of the material to be granulated comprising a spray drying granulation chamber (1) with substantially conical, downward tapering walls provided with

a drying gas inlet (2), and a liquid inlet (4) provided with an atomizer, arranged in the upper part of the chamber (1);

a drying gas outlet (3);

an inlet for a stream of solid feed particles (8, 11);

a material outlet (5) arranged at the lower part of the chamber (1) connected to a material inlet of a counter-current gas/gravity classifier (6) with a separation gas inlet (7), a product outlet (9) provided with a gas lock (14) and an outlet (8) for a fraction of fine particles suspended in separation gas connected to the inlet (8, 11) for the stream of solid feed particles.

13. An apparatus according to claim 12, wherein the material outlet (5) of the chamber (1) is connected to the material inlet of the classifier (6) via a gas lock (15), and the inlet (11) for the stream of solid feed particles is arranged in the upper part of the chamber (1) and connected to the fine material fraction outlet (8) of the classifier (6) via a conduit (10).

14. An apparatus according to claim 12, wherein a perforated horizontal plate for supporting a fluidized bed and an inlet for fluidizing gas (13) are arranged at the bottom of the chamber (1).

15. An apparatus according to claim 14, wherein an opening functioning as material outlet (5) of the chamber (1) and inlet (8) for the stream of solid feed particles is arranged at the lower part of the chamber (1) at a position over the perforated horizontal plate for supporting the fluidized bed.

16. An apparatus according to claim 14, wherein the material outlet (5) of the chamber (1) is arranged at the perforated horizontal plate for supporting the fluidized bed and connected to the material inlet of the classifier (6) via a gas lock (15) or as an opening arranged at the lower part of the chamber (1) at a position over the perforated horizontal plate for supporting the fluidized bed and connected to the material inlet for the classifier (6); and the inlet (11) for the stream of solid feed particles is arranged in the upper part of the chamber (1) and connected to the fine material fraction outlet (8) of the classifier (6) via a conduit (10).

Fig. 1

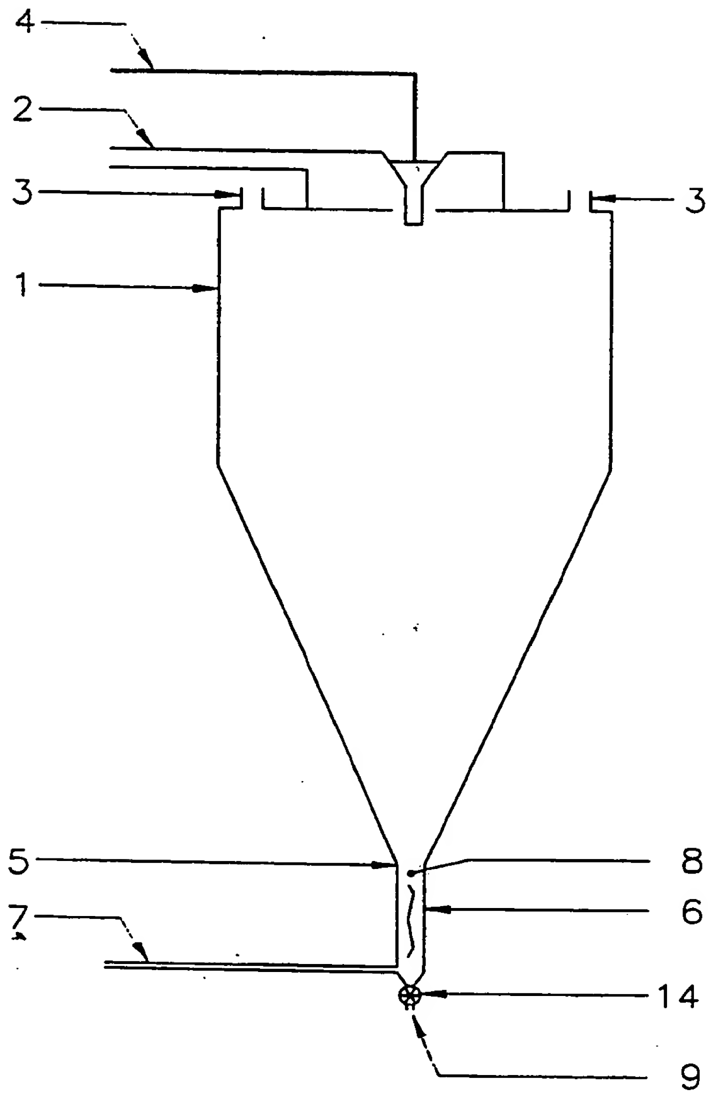


Fig. 2

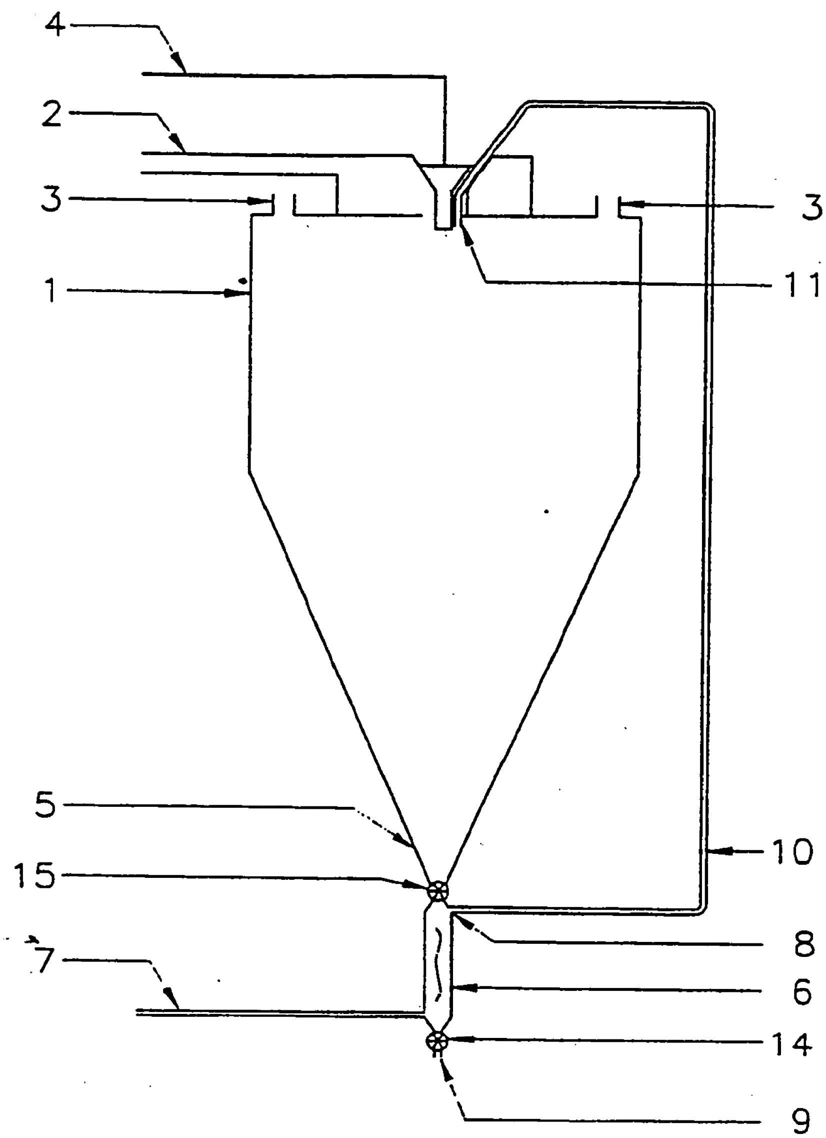


Fig. 3

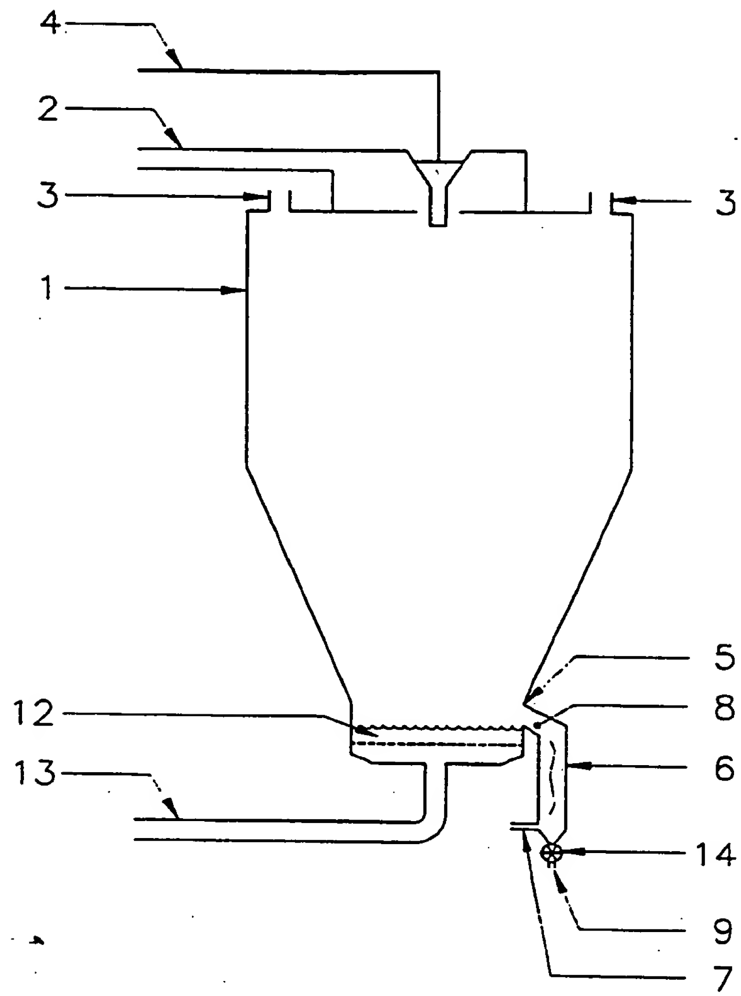
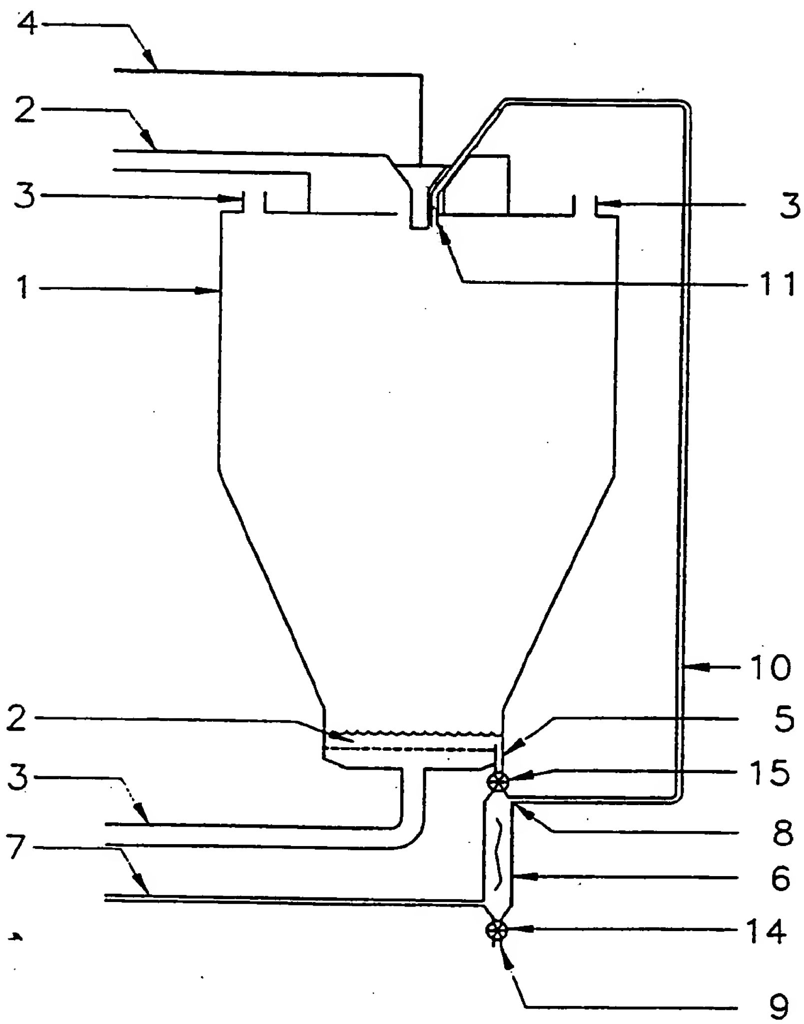


Fig. 4



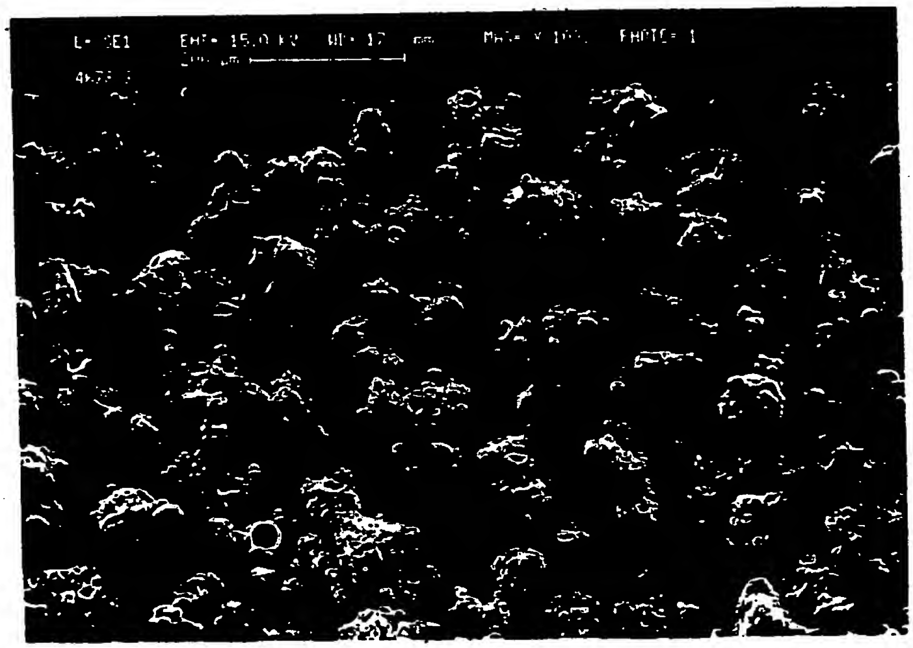


FIG. 5

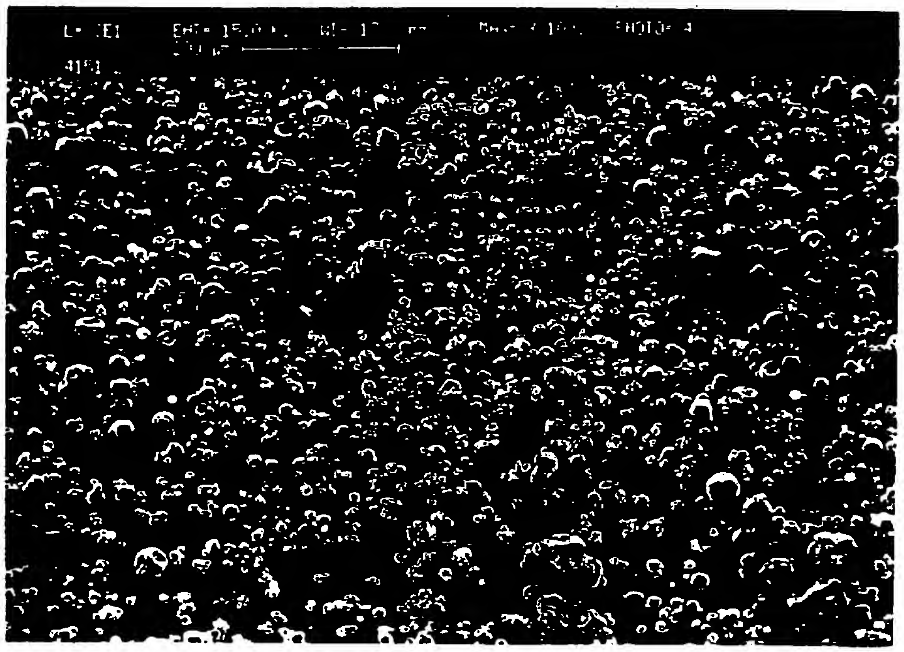
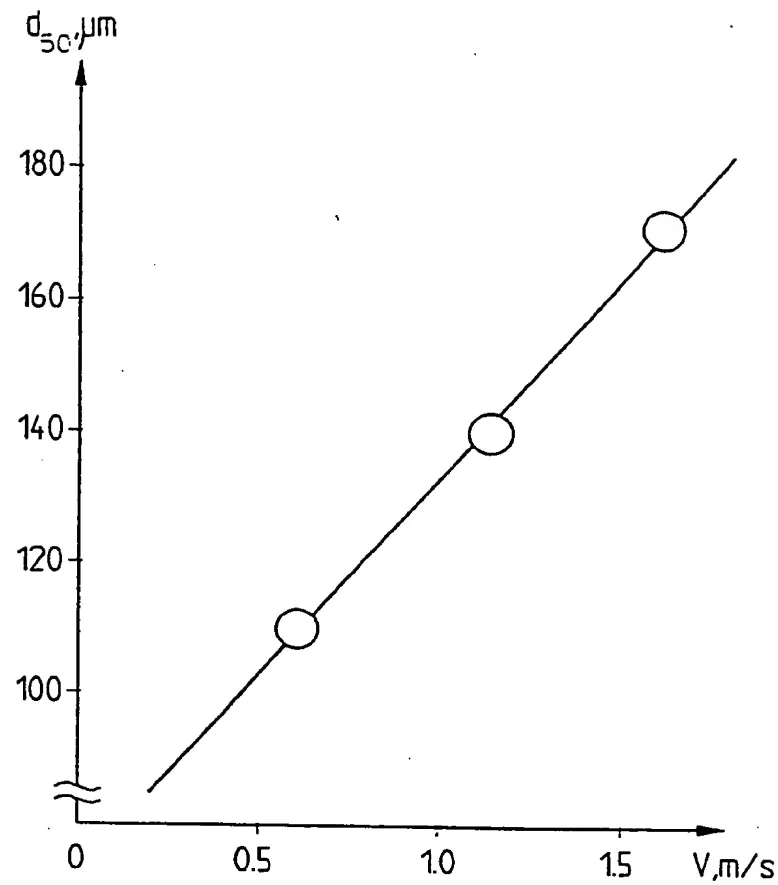


FIG. 6

Fig. 7



INTERNATIONAL SEARCH REPORT

International Application No PCT/DK 91/00043

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: B 01 J 2/04

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
IPC5	B 01 J; B 01 D

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched[8]

SE,DK,FI,NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category *	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
A	EP, A1, 0097484 (A/S NIRO ATOMIZER) 4 January 1984, see the whole document --	1,12
A	EP, A1, 0212714 (STAMICARBON B.V.) 4 March 1987, see the whole document --	1,12
A	US, A, 3735792 (NAOHITO ASIZAWA ET AL) 29 May 1973, see the whole document --	1,12
A	US, A, 3849233 (MIKHAIL V. LYKOV ET AL) 19 November 1974, see the whole document --	1,12

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* Special categories of cited documents:[10]

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
13th May 1991	1991 -05- 2 2
International Searching Authority	**Signature of Authorized Officer**
SWEDISH PATENT OFFICE	Eva Iversen Hasselrot

Form PCT/ISA/210 (second sheet) (January 1985)

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/DK 91/00043

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the Swedish Patent Office EDP file on 91-03-23
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